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Experiment number - 1 (Inorganic).

Object :— To prepare Ni - D.M.G. Complex.

Reagents :-

Nickel ammonium sulphate.	[3.8 g]
1% Alcoholic dimethyl glyoxime*.	[35 g]
1:1 Ammonia Solution.	[5 mL]
Absolute alcohal.	[5 mL]

Procedure :— Dilute the given solution of nickel ammonium sulphate to 150 mL in a 400 mL beaker by distilled water and add about 30-35 mL of 1% alcohalic dimethyle glyoxime solution. heat the solution of 70-80°C and then add 1:1 ammonia solution drop by drop with constant stiring till a distinct smell of ammonia perists long after. heat the beaker of a water bath for about half an hour. (This phenomenon is known as digestion.)

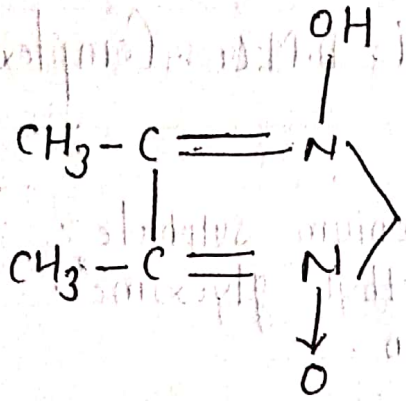
Allow the thick red precipitate to settle and test the clear supernatant liquid for complete precitation by adding few drops of the precipitating reagent.

i.e.- dimethyle glyoxime.

Reaction :— Nickel is precipitated as a complex salt on treatment with the alcohalic dimethyle glyoxime soultion in presence of ammonia.

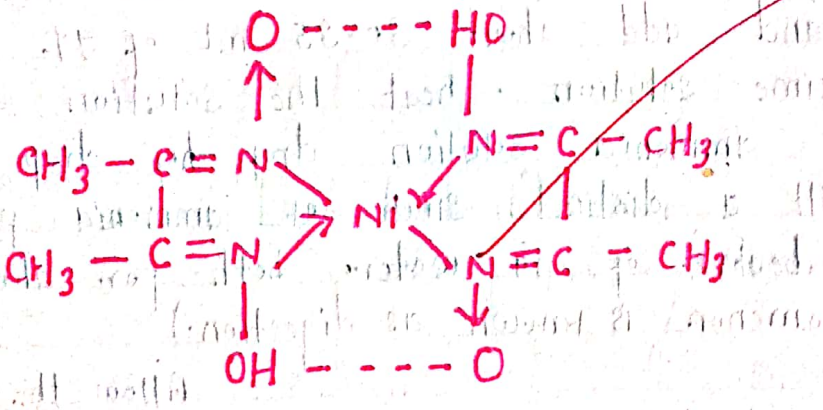
Teacher's Signature : .....

$$NiSO_4 + (CH_3C{=}NOH)_2 + 2NH_4OH \longrightarrow$$



$$Ni(C_4H_7O_2N_2)_2\downarrow + (NH_4)_2SO_4 + 2H_2O$$

Can your write the structural formula of Ni D·M·G Complex ?



Results :- 0.25 g.

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Experiment - 2.

Object :- To prepare tetrammine cupric sulphate.

Reagents :- 1- Copper sulphate $CuSO_4 \cdot 5H_2O$. 5g.

2. Concentrated ammonia (NH_3). 10 mL.
3. Alcohol (C_2H_5OH). 40 mL.

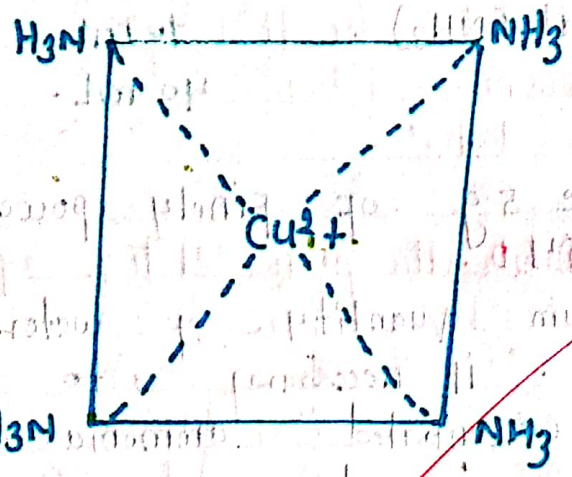
Procedure :- Take 5 g of finely powdered cooper sulphate $CuSO_4 \cdot 5H_2O$.

Dissolve it in minimum quantity of water adding a few drops of sulphuric acid. If necessary, to clear up the solution. Now add concentrated ammonia solution, with constant stirring from a dropping funnel, until the blue precipitate first formed completely dissolves to give a clear deep blue solution, and there persists a distinct smell of ammonia.

Reaction :- When copper sulphate solution is treated with concentrated ammonia solution and alcohal is added from a dropping funnel, tetramine cupric sulphate is obtained since it is insoluble in alcohal.

Results :- 5g.

long needle-shaped, blue-crystals of tetrammine copper sulphate seprated out. Filter and wash with a few drops of alcohal and dry in a desrecator.



dsp² - hybridigation
Square planar geometry.

submit your prepartion in a labelled tube (name of the preparation, its formula, seat no. yield etc.)

To the professor concerned for examination.

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Experiment number -3

Object :- To prepare sodium ferrioxalate.

Reagents :-

Ferric chloride ($FeCl_3$) 10 g.
Sodium hydroxide ($NaOH$) 11 g.
Oxalic acid ($COOH \cdot COOH$) 12 g.

Procedure :- First of all ferric oxide is to be prepared from ferric chloride.

preparation of hydrated ferric oxide. 10g of $FeCl_3$ is dissolved in 5 mL of water and to it sodium hydroxide solution. (7.5g NaOH in 5 mL of water.) is added in small quantities. Now filter the precipitate of $Fe(OH)_3$ and wash it several times with small quantities of hot water.

preparation of Sodium Ferrioxlate -
Now 12g oxalic acid is dissolved in 50 mL of hot water and to it 4g of NaOH (pellets) are added in small quantities. Now hydrated ferric oxide is added to this hot solution until it is in excess. The solution is constantly stirred with a glass rod.

Now the solution is filtred. the green filtrate is constanly stirred with a glass rod. Now the solution is filtered. the green filtrate is concentrated in silica dish on a water bath to crystallization. green crystals of

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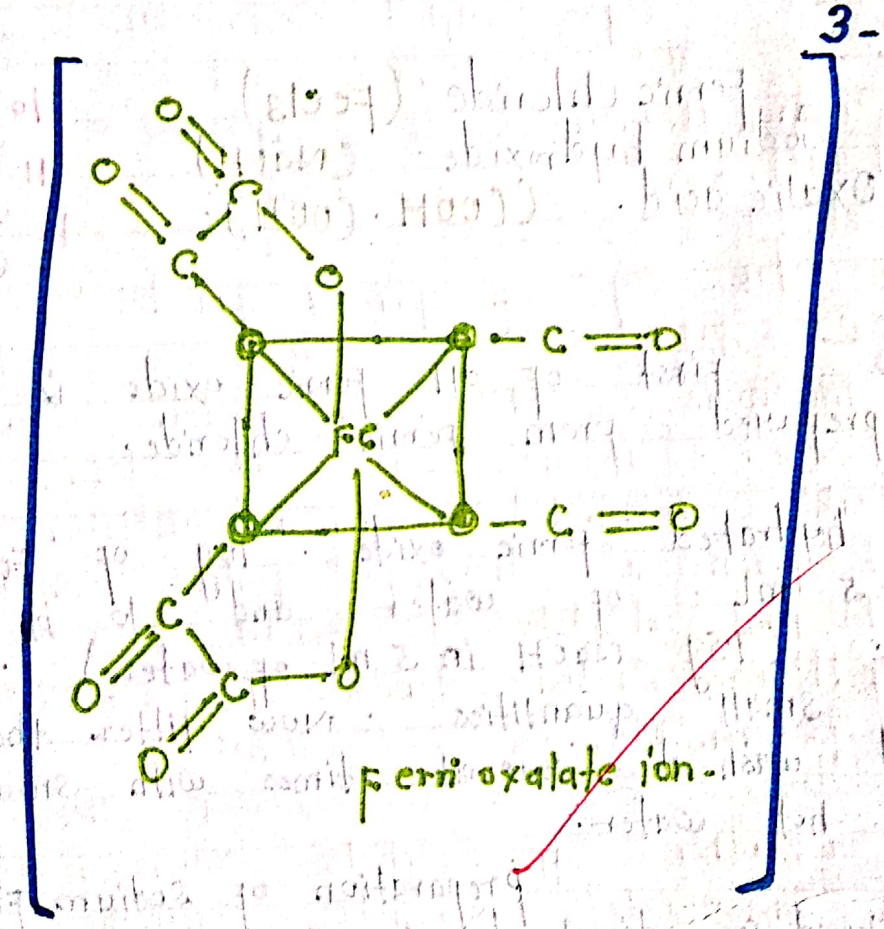

ferri oxalate ion.

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Sodium ferrioxalate are formed.

Submit your prepraation in a labeled tube (Name of the prepration, its formula, Seat No yield etc.).

To the professor concerned for examination.

Results :- 15 g.

Structure here Fe(III) is bound to 3 bidenate oxatato ligand. Coordination number of Fe(III) is 6 and it is octahedrally srounded by six orgen atoms - two each from 3 oxalato group.

paramagentic with 5 upparied electrons. this can-be explained on the basis of sp^3d^2 hybridization of Fe(III) ion.

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Experiment number-4

Object :- To prepare cis and trans potassium bioxalato diaquo chromate (III) dihydrate.

Reagents :-

Potassium dichromate	2g
Oxalic acid (Hydrated)	6g
Ethanol	40 mL

Procedure :- Take 2g $K_2Cr_2O_7$ and 6g $H_2C_2O_4 \cdot 2H_2O$ in dry mortar*. Grind the mixture gently to fine powder. now take this mixture in a china dish and add few mL water to moisten it. Cover the dish with clock glass and heat very slowely**.

After few minutes a vigorous reaction takes place with forthing due to the liberation of CO_2 gas and water vapours. Thus the mixture changes to deeply coloured liquid***. pour 20 mL ethanol over this hot, mixture.

It is stired with metallic spatula. if complete solid is not obtained add 10 mL. ethenol more. warm it to get granular crystals these crystals look like black in diffused day light while deep purple in artifical lighit.

Submit your prepration in a labelled tube (name of the preparation, its formula, Seat No, yield etc.).

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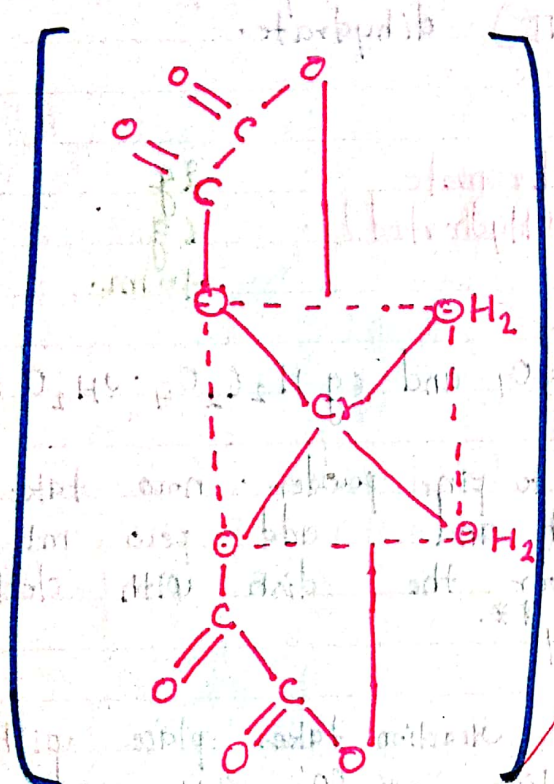
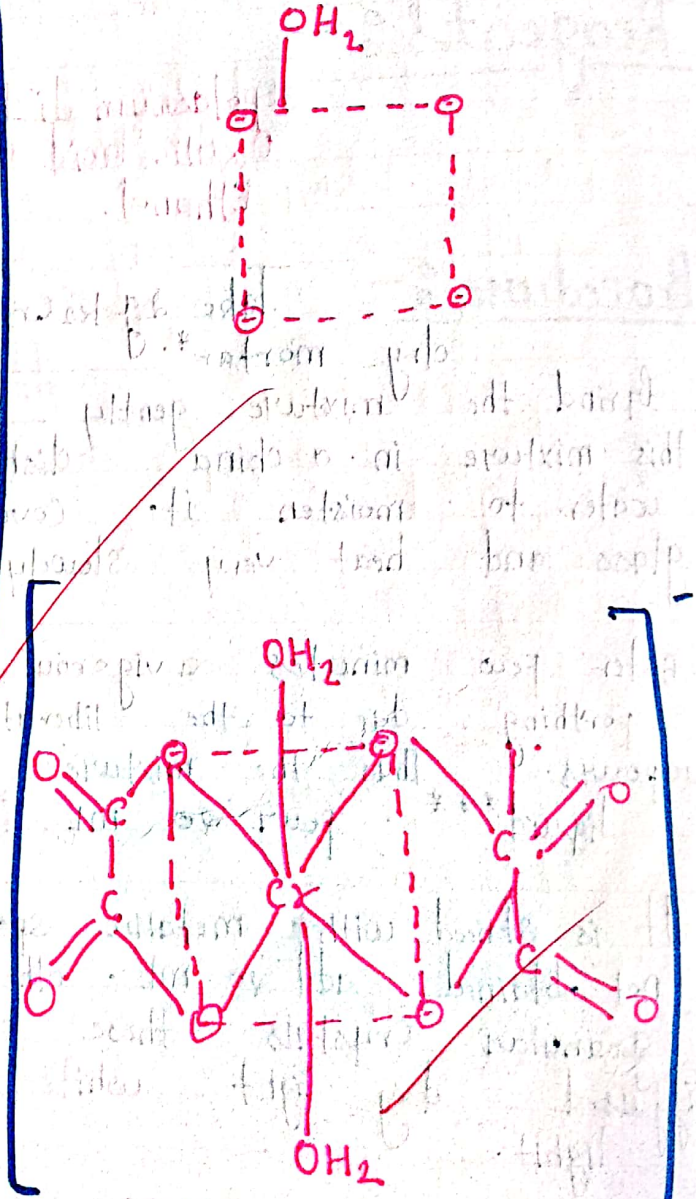


Cis form



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To the professor concerned for examination.

Reaction:- The complex may be obtained by the reaction of potassium dichromate and oxalic acid.

$$K_2Cr_2O_7 + 7H_2C_2O_4 \cdot 2H_2O \longrightarrow$$

$$2K[Cr(C_2O_4)_2(H_2O)_2] \cdot 2H_2O + 6CO_2 \uparrow + 13H_2O.$$

Results:- 3g.

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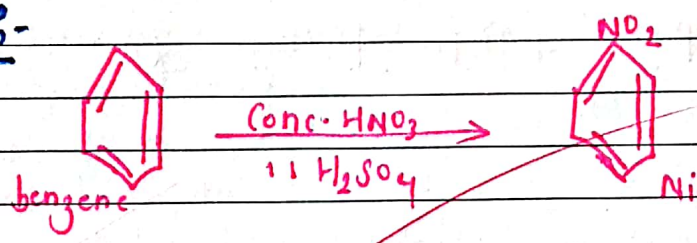
(Organic — Compound):— Experiment Number — 5.

Object :- To prepare nitrobenzene from benzene.

Reagent :- benzene 10 ml . Conc . HNO_3 11.5 ml . Conc. H_2SO_4 13.3 ml.

Procedure :-



In a 100 - 150 ml round bottom flask take 11.5 ml conc. HNO_3 (Sp. gr. 1.4), then add, in small portions at a time with continuous vigorous shaking, 10.0 ml of Conc. H_2SO_4. during mixing the acid, cool the flask in ice old water. now introduce 10 ml of benzene (in portions of 0.5 to 1.0 ml). with continuous shaking the flask to ensure through mixing. the temperature should not be allowed to rise above 55 - 60°c.

Immerse the flask, if necessary in, cold water; when all benzene has been added heat and attach with the condenser. (Condenser of 5" long will be sufficient). during heating shaking of the flask time to time will also insure good mixing of the immiscible layers.

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now cool the contents and pour in 250 c.c ice-cold water containing crushed ice. taken in a beaker. Stir the mixture well to wash out as much acid as possible from nitro benzene and allow the stand. when nitro benzene has settled down at the bottom power off the upper aqueous layer as completely as possible and transfer the residual liquid to a separating funnel. Separate the lower layer of nitro benzene and reject the upper aqueous layer. Again return nitro benzene to seprating funnel and shake it vigorously with 20 ml of water.

Now run out the lower layer of nitro benzene into a conical flask cantaining about 2g of anhydrous CaCl2. Shake the mixture (warm it if necessary.). until the clear liquid is obtained. The yellow liquid is transfered by decantation to a 50 ml distillation flask fitted with an air condenser.

Heat the flask on an substance centred. grauze or an air bath and collet the fraction distilling between — 205 - 210°C.

Result:- Yield 10.6 g,
9.0 ml.
B.P. 210°C.

Appearance:- pale yellow liquid.

Teacher's Signature : .....

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Experiment number - 6

Object :- To prepare p- nitro acetanilide from acetanilide.

Reagents :- Acetanilide 10.0 g, Glacial acetic acid 10.4 ml, fuming HNO_3, 4.4 ml, Conc. H_2SO_4 20.0 ml.

Procedure :- Dissolve 10.0 g of acetanilide in 10.4 ml of glacial acetic acid. taken in a conical or round bottom flask.

(Warm the contents if necessary to get a clear solution.)

Now and 20.0 ml of Conc. H_2SO_4 gradually with vigorous stirring. (The solution will become hot).

Cool the flask in freezing mixture of ice and salt and stir the contents will now add from drop by drop with regular stirring, 4.4ml. fuming HNO_3 to the flask placed in ice bath and temperature is maintained below 10°c. when all the acid has been added allow the mixture to stand at room temprature for half an hour. pour the reaction mixture on to 100 g of crushed ice - where by the crude nitro acetanilide is precipitated

The product is diluted with 100 ml of cold water and allowed to stand for 10 minutes. o-Nitro acetanilide is precipitated the product is diluted — (a little amount formed.) goes into the solution while p-isomer remains insoluble. filter it at pump, wash thoroughly with cold water

Teacher's Signature : .....

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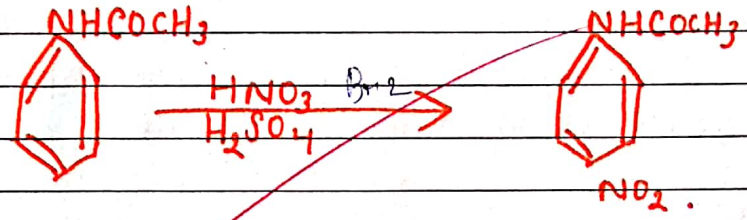
and drain well. recrystallise the pale yellow product from alcohol.

Reaction :-



Result :- Yield 8.0 g. M.P. 244°C.

Appearance :- Colourless crystaline compound.

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Experiment number - 7

Object :- To prepare picric acid (2:4:6 - trinitrophenol) from phenol.

Regents :- phenol 7.5 g . Conc. - H_2SO_4 9.8 ml , Conc. Nitric acid HNO_3 . 28.5 ml.

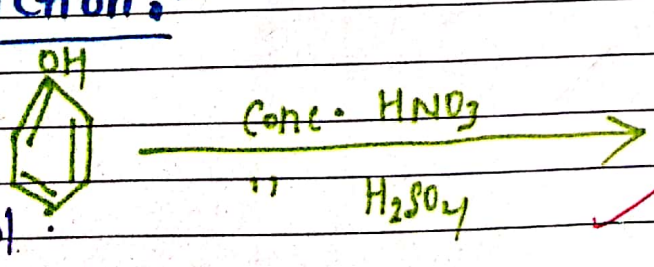
Procedure :- In a round bottom flask take 7.5 g of phenol and 9.8 ml conc. H_2SO_4 , shake and heat on a water bath for about 30 minutes. Cool the flask in an ice bath to about 40°C and then place it on the table.

add 28.5 ml of conc. HNO_3 slowely with stiring brown fumes of NO_2 gas are formed in excess. when this reaction subsides , heat the flask on a water bath for about 3 hours.

with occasional shaking . Cool the flask and then pour the contents of this flask into 400 ml of water .

A yellow solid product seprates from out it. is filterd washed with water , recrystallised from dilute alcohal and dried.

Reaction :-



phenol . picric acid.

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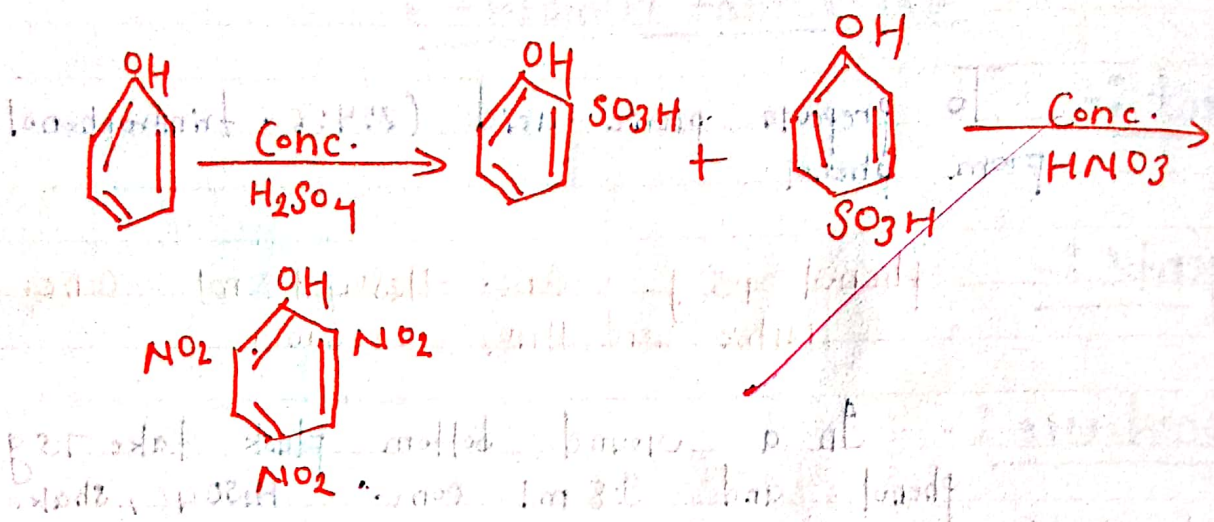

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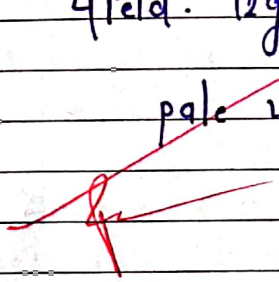
Result:- Yield. 12g · M.p. 122°C.

Appearance:- pale yellow leaflets.



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Experiment number - 8

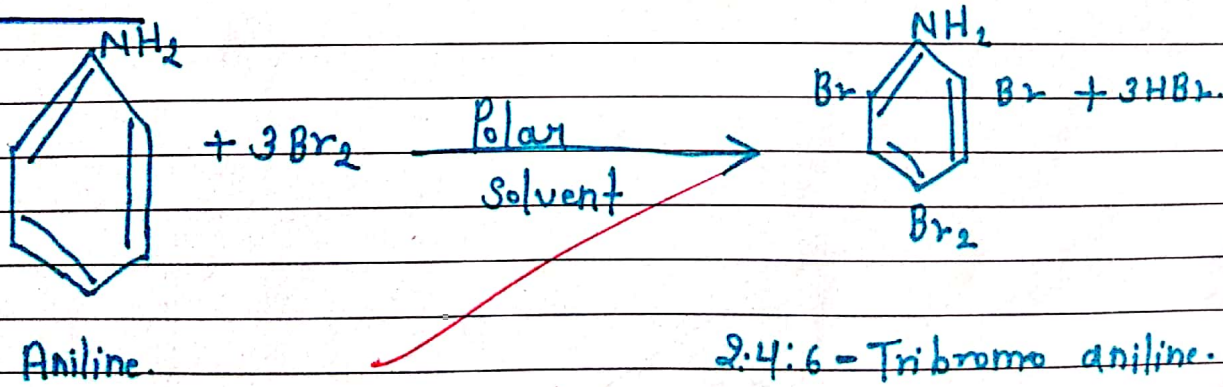
Object :- To prepare sym · 2:4:6 tribromo aniline from aniline.

Reagent :- Aniline 5·0 ml, Glacial acetic acid 40·0 ml bromne 8·5 ml (27·0g).

Procedure :- In a 250 ml Conical flask, 5·0 ml of aniline in 20·0 ml of glacial acetic acid. mix 8·5 ml of bromine with another 20·0 ml of glacial acetic acid and take this solution in a dropping funnel placed over the flask · now add the bromine solution dropwise with constant shaking the flask To the aniline solution · (during addition of bromine, The flask should be cooled in ice cold water as the reaction is exothermic.).

A yellow coloured paste would be obtained · (If the colour is not yellow then add a little more bromine solution.).

Reaction :-



Aniline. 2:4:6 - Tribromo aniline.

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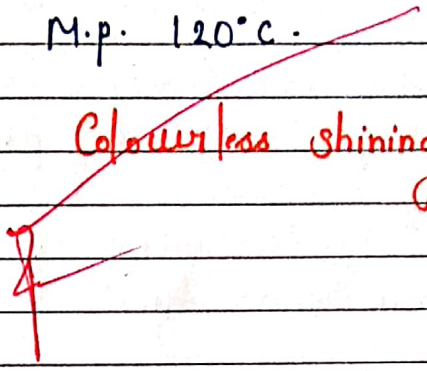
Result :- 12.0g. M.p. 120°C.

Appearance :- Colourless shining needles.



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Experiment number - 9

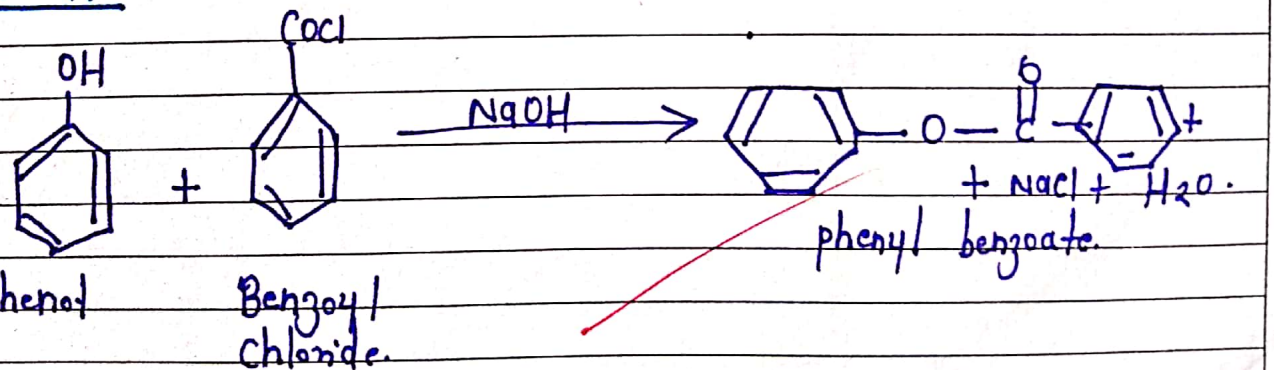
Object :- To prepare phenyl benzoate from phenol or benzoylation of phenol.

Reagents :- phenol 2 g, benzoyl chloride 4 ml, Aq. $NaOH$ (10%) solution 30 ml.

Procedure :- Take 2 g phenol in 250 ml flask and add 30 ml of 10% aqueous solution of NaOH. now add 4 ml. benzoyl chloride cork the flask and shake vigorously for about 30 mintues. crude phenyle benzoate seprates as a white solid. reaction is completed when the smell of the benzol chloride can no longer be dedacted. Filter the product obtained wash with cold water and drain well.

crystallise the product from ethyle alcohal. Filter at pump, wash throughly with alcohal and dry.

Reaction :-



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Result :- 3 g M.P. 68°C.

Appearance :- Colourless crystals.

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Experiment number- 10

Object :- To prepare benzoic acid from toluene.

Discussion :- benzoic acid is obtained by the oxidation of toluene. (by HNO_3, acidic $KMnO_4$ etc.).

$$C_6H_5 \cdot CH_3 + 3[O] \longrightarrow C_6H_5COOH + H_2O$$

Toluene — benzoic acid.

Reagents :- (i) Toluene — 10 g
(ii) Aqueous solution of $KMnO_4$
(iii) Dil. H_2SO_4.

Procedure :- mix 10 g toluene and few millilitre of dill. H_2SO_4 till light pink colour appears. heat the mixture of some time. the nascent oxygen formed by the reaction of $KMnO_4$ and dill. H_2SO_4 oxidises toluene into benzoic acid at the pump. wash it with a little cold water and recrystallize from 150-200 ml boling water. benzoic acid is obtained as colourless crystals.

Signed 03/01/19

Teacher's Signature :

Observations :-

क्रमांक	प्रयुक्त NaOH का आयतन मिलि में	चालकता Cmho
1	–	–
2	–	–
3	–	–
4	–	–
5	–	–

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Physical :- Experiment number - 11

Object :- To determine the strength of acetic acid by titrating it against standred NaOH solution conductometrically.

Apparatus :- Conductivity bridge, burette, pipette measuring flask, NaOH, acetic acid oxalic acid etc.

Theory :- It is already described in previous experiment for determining the strength for the acid or alkil, the following formula is used.

$$N_1V_1 = N_2V_2.$$

Method :- The unknow solution (10ml) of acetic acid is taken in a 100 ml beaker and the conductivity cell is dipped in this solution. NaOH solution is first of all standard dised against standred oxalic acid solution and then filed in a micro burette. the conductivity bridge is connected to battery or electricity and 0.1 ml of NaOH is added from burette in the beaker, the solution is stricred and its conductivity is determind. repeat the experiment by adding 0.2 ml - 0.3 ml - of NaOH until the break in the curve has been observed.

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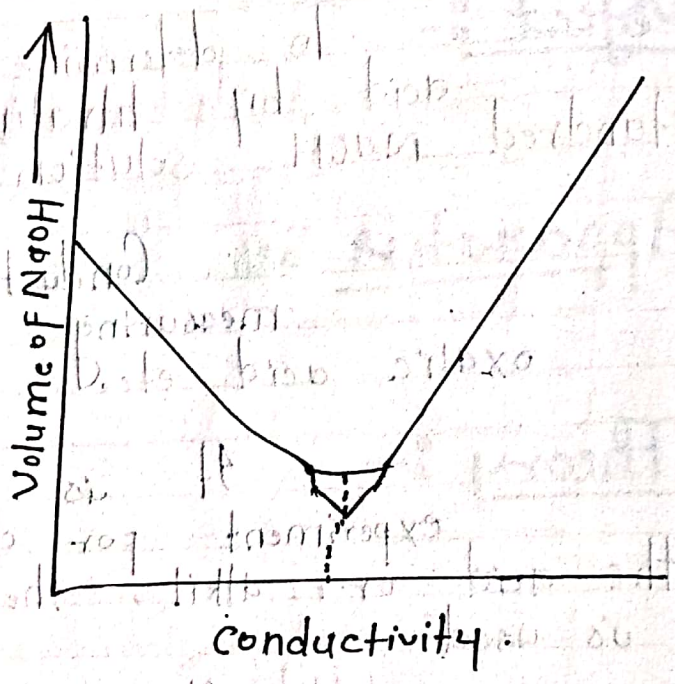
Calculation :-



Acetic acid = NaOH

$N_2 \times 10 = N_1 \times \alpha$

$\therefore N_2 = \frac{N_1 \times \alpha}{10}$

∴ Strength of acetic acid in gms/liter.

$= \frac{60 \times N_1 \times \alpha}{10}$ gms/liter.

(where $N_1 = 60$ equivalent weight of acetic acid).

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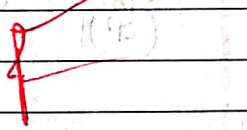
From graph the volume of NaOH used in (calculated by drawing perpendicular on Y axis from the point of intersection) = x ml.

Result:- The strength of acitic acid = ... gl^{-1}.



S.No.	Position of N_2 with H_2O in tube		position of N_2 with sugar solution in tube		Angle of rotation.		
	Clock wise (a)	Anticlock wise (b)	Clock wise (a')	Anti Clock wise (b')	$(a'-a)^\circ$	$(b'-b^\circ)$	Average digree.
1.							
2.							
3.							

Calculation :-

Formula for calculation is -

$$[\alpha]_D^t = \frac{\alpha_{obs}}{l \cdot c.}$$

by putting the value for α_{obs}, l and c $[\alpha]_D^t$ can be calculated.

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Experiment number- 12

Object :- To determine the specific rotation of a given optically active compound (Sugar).

Apparatus :- Polarimeter (bi Quartz) Sodium lamp measuring cylinder, beaker, Physical blance etc.

Theory :- The specific rotation of the given compound may be given as —

$$[\alpha]_D^t = \frac{\alpha_{obs}}{l \cdot c}$$

All the terms have usual significance.

Procedure :- Fill up the polarimeter glass tube with pure water. there should be no air bubbles in the tube. now place is at its proper place. Switch on the bulb and direct the lens L of the polarimeter towards the bulb and look the light through eye peice, E. Two haules of diffrent colours will appear in it. now rotate analyer N_2 slowely in such a way that exactly same couler is observed in both the haules, note down this reading of N_2 in circular scale. now rotate N_2 in other side to get the exactly same colour and note this reading also.

prepar 10% solution of Sugar by dissolving 10 g sugar in 100 cm^3 water. Remove the water in

Teacher's Signature : .....

Observation :- (A) mass of empty weighing tube = $w_1 g$.

mass of weighing tube + Sugar = $w_2 g$.

mass of Sugar = $(w_2 - w_1) g$.

Volume of water = $V cm^3$

$\therefore$ Concentration of Sugar Solution = $\frac{w_2 - w_1}{V} = c g\ cm^{-3}$

(B) Room temperature = $t^\circ C$

(C) length of polarimeter tube = l decimeter.

(D) Table for angle of rotation

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polarimeter glass tube and fill up this solution of sugar. (Remove air bubble in the tube if any). and place it's proper place. rotate the analyser N1 to get the same colour two halves in eye piece in both. (i.e., clockwise and anticlockwise).

direction similar to water. note down these two readings for both these position. this difference of this reading and that water is the angle of rotation, 'a' for the concentration, measure the length 'l' of the polarimeter glass tube in decimeter.

Result :- The specific rotation of given substance (sugar) at _____ °C is _____

od/m gcm-3.

Hamood
05/01/19

Teacher's Signature : .....